DOE-S&A FY03 ANNUAL REVIEW MEETING

PHASED Feasibility Demonstration[1]

(Phased Heater Array Structure for Enhanced Detection)

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(1) "Industrial Wireless PHASED Sensor. Phase 1. Feasibility Demonstration," Final Report to DOE/ORNL, Contract DE-AC05-00OR22725, Honeywell Labs, Plymouth, MN, 30 April 2003

(2) with contributions by E.Satren, S.Eickhoff, H.Pham, J. Detry, S.Swenson, L.Hilton, R.Nickels, G.Swenson, C.Cabuz, A.Barron

Project Overview of PHASED µAnalyzer

- **Project description**
  - PHASED is to fill the need for faster, smarter and more affordable chemical composition analysis devices (µAnalyzer). It is based on Si-micromachined channels with integrated flow, temperature and TC sensors, and featuring multi-stage PC and electronic injection.
- **Objectives**
  - Provide a bench-top feasibility demonstration of PHASED.
  - Complete chip design and layout; processing of heater arrays and adsorber film material; test and drive electronics
  - Demonstrate multi-stage pre-concentration, e-injection and separation
  - Recommend future work
- **Overall goal**
  - Develop a self-contained, palm-top-sized, hi-speed, hi-sensitivity µanalyzer, which is to be compatible with wireless and NeSSI

Technical Merit of PHASED

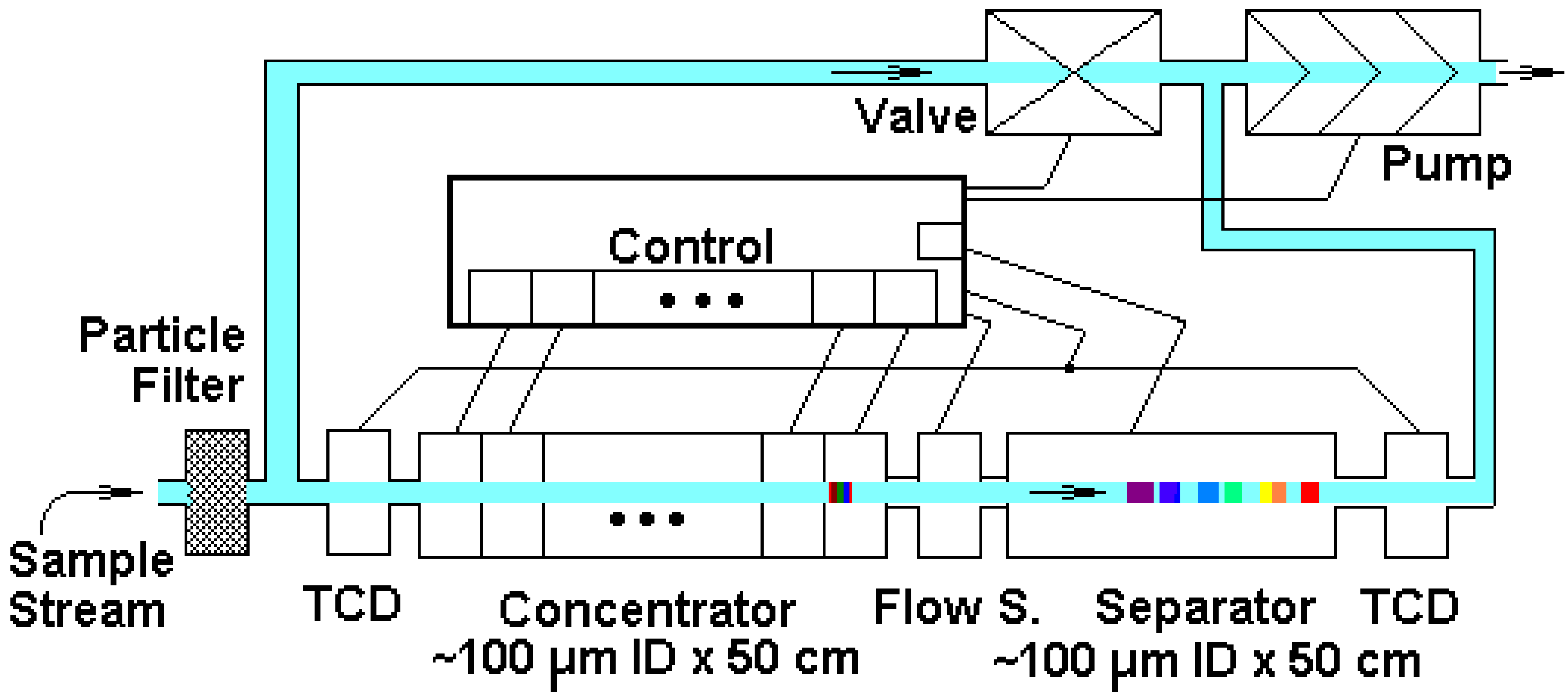
- **Addresses technical need(s) of the S/C community and the S/C priorities of the IOFs**
  - The IoF Reports state the need for more affordable and reliable chemical analysis devices, to better control the process i.e. energy, product quality and emissions
  - IoF beneficiaries would be the chemical, petrochemical, pharma, food industries as well as the metals processing industries. Some examples: Ethylene plant process stream composition, methyl-iodide emission, impurities in H2 generation streams

Technical Merit of PHASED

- **Contributes new information or technology to the S/C community**
  - First step towards a NeSSI-compatible microanalyzer/GC
  - Leapfrogs GC analyzer technology capabilities: relative to GCs
    * with only one pre-conc. stage 100x more sensitive
    * without any pre-concentration 1,000-10,000x more sensitive
  - Composition analysis within 1-2 seconds, or 10-100x faster than conventional GCs
  - More affordable: 10-100x lower cost

Technical Progress and Outlook of PHASED

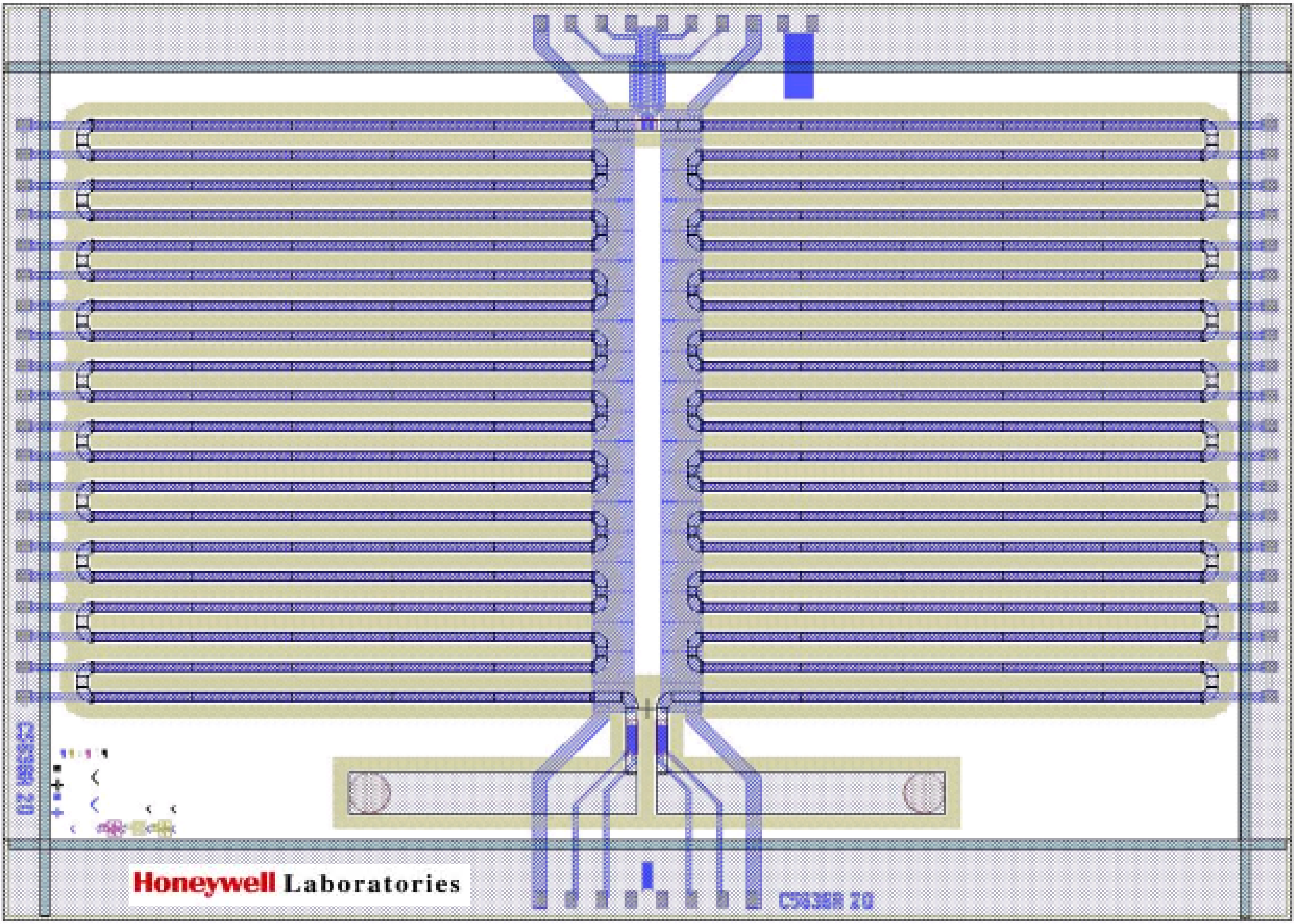
- **Provide bench-top feasibility demonstration**



Technical Progress and Outlook of PHASED

- **Completed chip design/layout/fabrication**
- **Completed drive and test electronics**
- **Demonstrated multi-stage pre-concentration, e-injection and separation**
- **Predicted PHASED performance via math simulation**

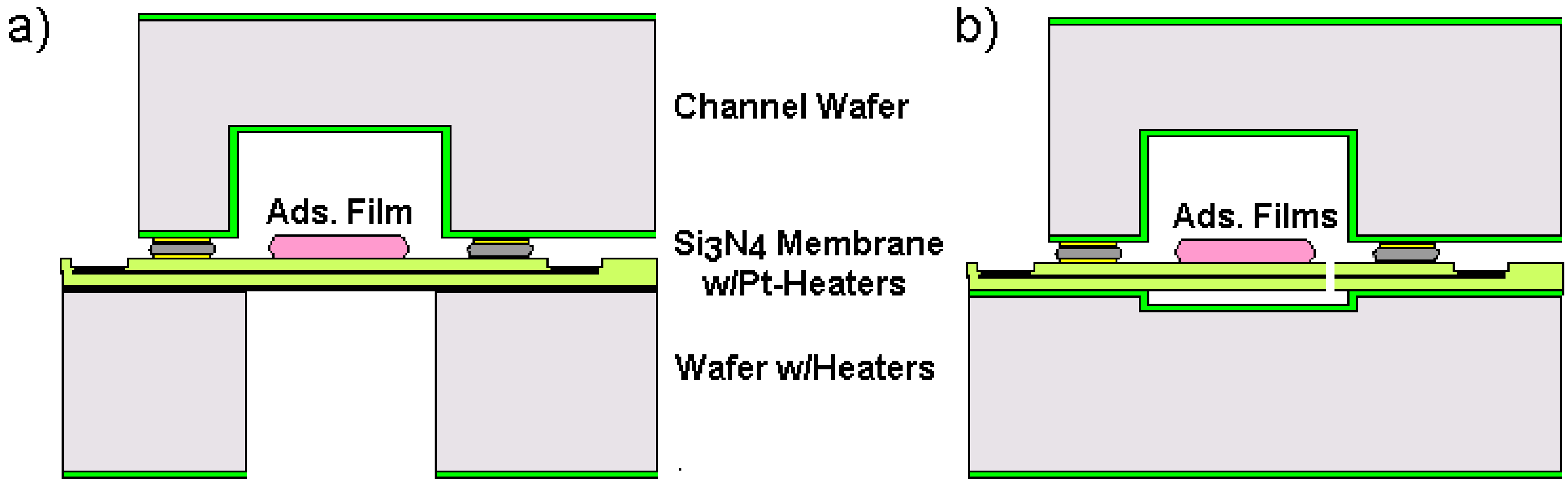
as shown in the next 6 VGs



20-Element Pre-Concentrator, Diff. TC, 20-Element Separator

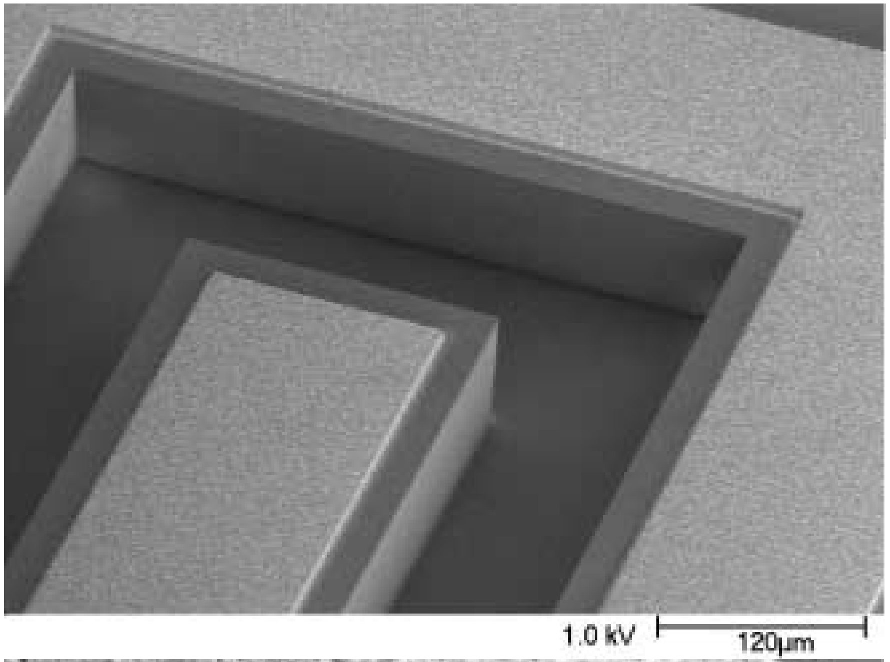
Integrated Version of PHASED, Showing Sensors, Concentrator and Separator.

Technical Progress - PHASED



PHASED Micro Analyzer Views:

a) Cross sectional view of PHASED-I/-II with exposed 1-µm membrane
b) Cross sectional view of ruggedized and low-power PHASED-III



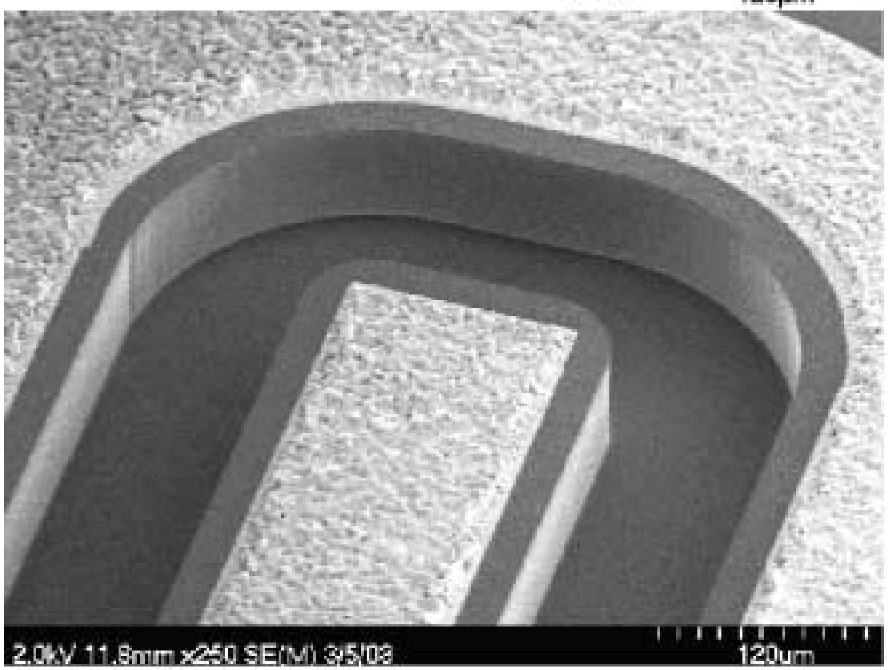


Fig. 3. Underside of PHASED Sensor Chip, with Heatable Elements in Each of the 2 x 20 Etched Grooves Visible in the Photograph.

SEM-Graph of PHASED Run #2 Chip Showing 1) Rounded Channels Etched via DRIE and 2) Wafer-Wafer Bonding and Channel-Sealing Metal. 5-Mar-03

Honeywell Laboratories

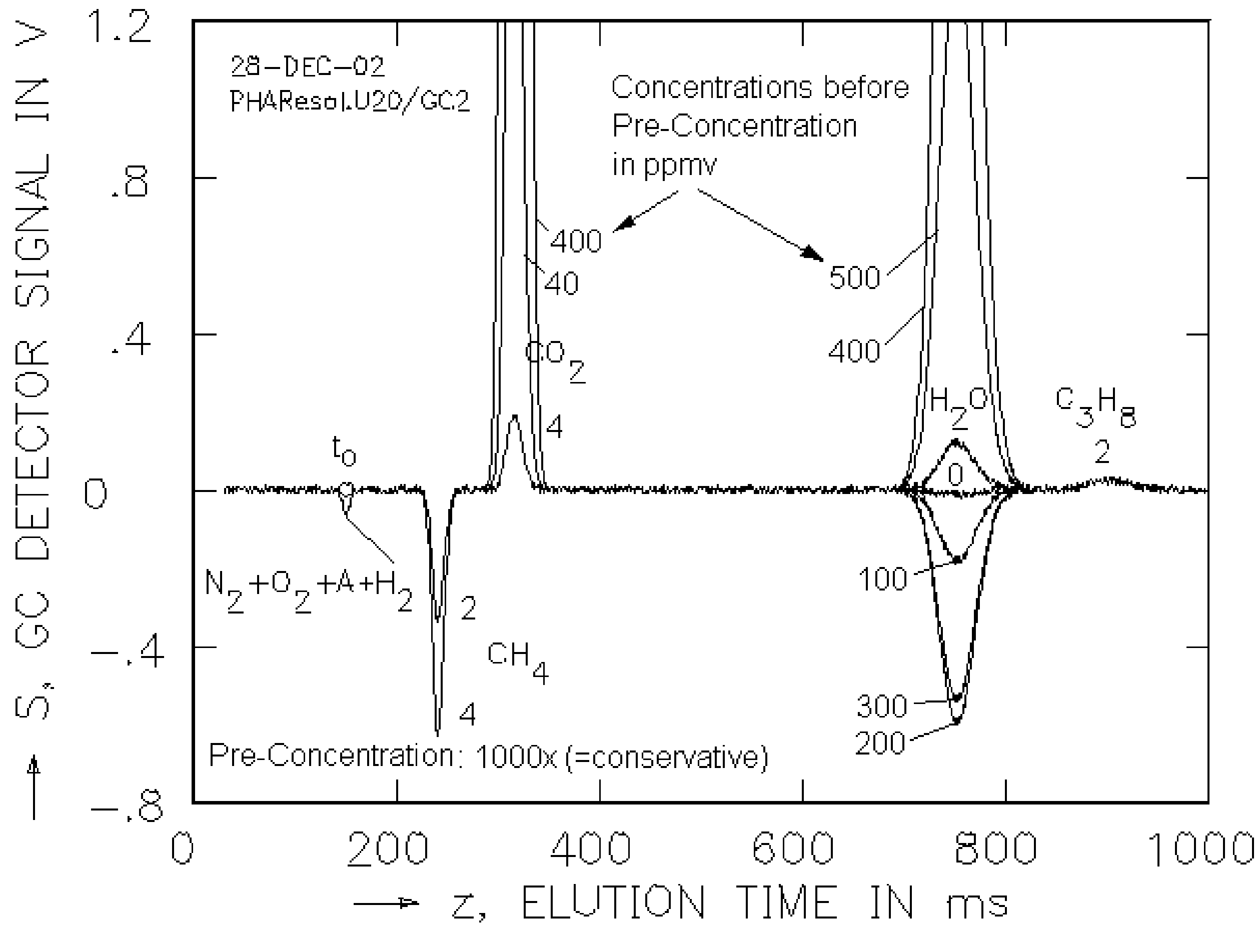
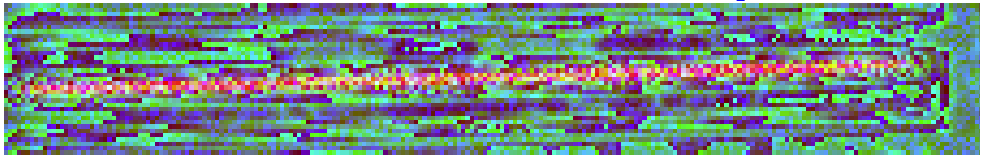
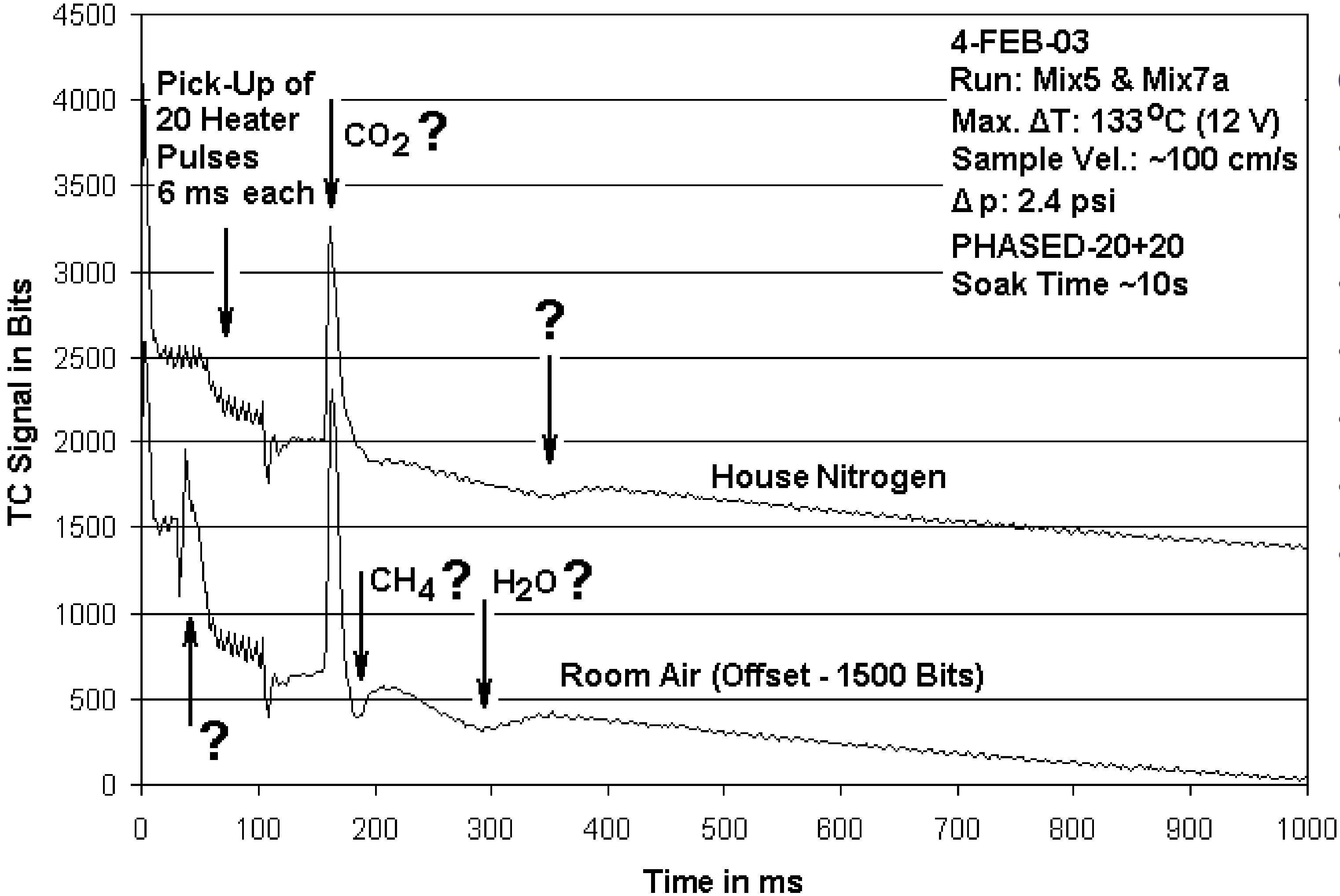


Fig. 32. Simulated PHASED Outputs for the 50−Elem. Version with L=25 cm, v=166 cm/s, R=15. Thermal Conductivity Detector at 100°C. Conc. Gain: 1000x; NEC(CH_4): 0.2 ppm−PP.

PHASED: 1st Exp'tl. Output for N2 & Air

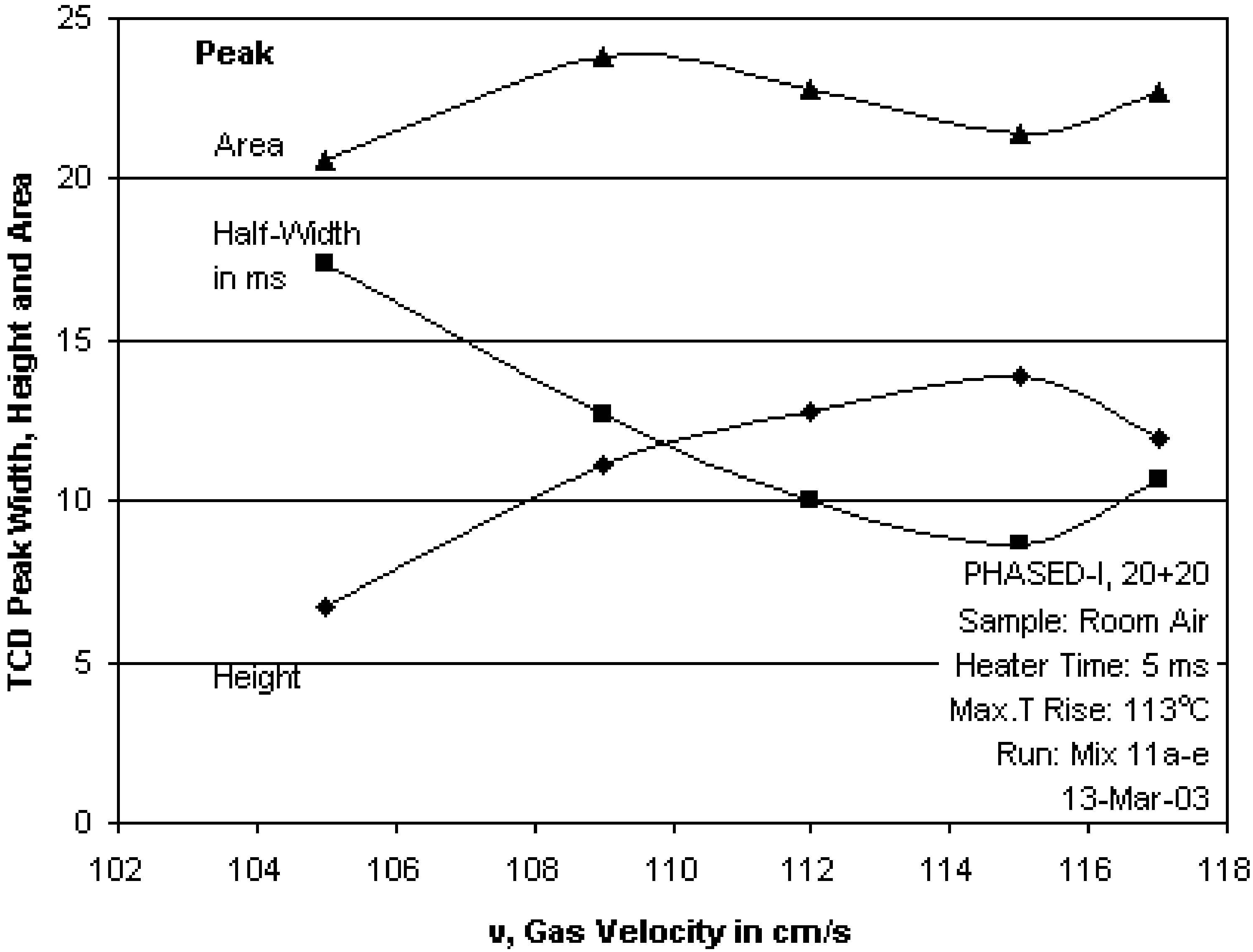


Demonstration of Uniformity and Localized Heater Pulse with IR Camera



Questions:

- **Peak ID**
- **Leakage**
- **Calibration**
- **Opt. Synch**
- **Sens.Limit**
- **Pre-Concen.**
- **Resol.w/50+50**



Sensitivity of PHASED Output to Proper Synchronization between Rate of Gas Velocity and Rate of Heater Array Progression. Minimum Half-Width is Near 8 ms, i.e. Excellent Compared to 5 ms Heater Pulses. Run # 1. 20+20-Elements. 13 March 2003.

Technical Progress and Outlook of PHASED

Measured Performance and Feasibility Demonstration:

1. **Time:** Analysis is as short as predicted: ≤ 1-2 seconds
2. **TC**: Peaks above (CO_2?) and below (CH_4, H_2O?) the base line as predicted, due to their TC values relative to that of air
3. **Analytes**: Eluting peaks are different between house-N_2 and room air. In room air we see one small but repeatable peak that could correspond to the ~ 2 ppm CH_4 of normal air

3. **Flow:** The elution time of these analytes is flow rate-dependent as it should

4. **Synchronization**: between rate of gas velocity and rate of heater array progression. We observed:

- Peak half widths, W~8 ms, i.e. consistent with the 5 ms heater pulses, and indicating very satisfactory stacking of 20 pre-concentration stages
- Flow velocity mismatch exerts a strong influence on W and peak height, H: W and H change by 2x for only ~8% change in flow velocity.

Technical Progress and Outlook

Future Technical Milestones/Goals

Milestone/Goal	Expected Completion Date	Comments
Complete evaluation of PHASED vs. peak identification, GC-film material and thickness, temperature programming, stability, sensitivity limits, analyte range		
Ruggedize PHASED, and flow control designs		
Consider alternatives to TCD to add flexibility and sensitivity		
Upgrade electronics, MMI, wireless		
Field test and commercialization		
Project Completion	~ 2 years	

Technical Progress and Outlook

- **Expected progress toward milestones/goals**
  - Achieve the listed Future Goals within ~ 2 years
- **Possible barriers or risks**
  - PHASED chip yield is low
  - Available pumps and valves are not reliable enough
  - The available TCD is not sensitive enough

Technical Progress and Outlook

- **Industrial end-user involvement**
  - Several CPAC* members from the chemical, petrochemical, food, semiconductor and metals industries are interested in and intend to field test PHASED prototypes.

  * CPAC = Center for Process Analytical Chemistry

Market Potential of PHASED

- **Commercialization plan**
  - R&D: Complete at Honeywell Labs, w/inputs from SBUs
  - Technology Transfer Path: Field tests at CPAC Members' sites; open architecture
  - New Products Intro to Industry Cross-Section: Honeywell has established channels to market across IoFs. Sensors and process automation controls, in addition to service and operation contracts
  - Partnership Strategies: Long-term partnership with industrial processors for service and plant operations

Market Potential of PHASED

- **Other IOF areas of applicability**
  - General “chemical composition” microanalyzer for measuring emissions, process stream composition and impurities, and product quality

Market Potential of PHASED

- **After OIT project (Phase I: Feasibility Demo), what's next?**
  - Technical: Prepare and submit proposal for Phase II to evaluate performance and limitations; ruggedize structure; design and fab NeSSI compatible version; field test at IoF/CPAC Members' sites
  - Marketing: Evaluate market potential, and plan productization accordingly (make, partner, license,…), for broadest applicability
  - Productization: Gear up to manufacture, market, sell, and service

Programmatic Merit of PHASED

- **Energy benefits** estimated by:
  - Identifying specific examples of processes
  - Quantifying their energy reductions assuming that analyzer platforms will be deployed to all U.S. industrial processors
  - Using energy reductions from these examples and extrapolating to other applications in the same IoF group
  - Inserting these numbers into the DOE Savings Calculator

The specific IoF examples account for 40% of the energy consumed by U.S. industry*.

	TBtu/y savings for 2010	2020
– Ethylene production (petroleum refining) - 2.06%	2.1	18.3
– Hydrogen production (petr.ref. & chemicals) - 0.83%	0.52	5.35
– Metal treating (prim.metals & fab. met.parts) - 10%	2.5	16.2
– Fired heater control (petr.ref. & petrochem.) - 1.3%	10.3	18.2

* Energy Information Administration, “Manufact.Consumpt.of Energy '98”, Energy Use Data, Table N1.2, as referenced in Honeywell’s Proposal to DOE**

** U.Bonne et al, “NeSSI-Compatible Microanalytics,” proposal to DOE-S&C, 16-Apr.-03

Programmatic Merit of PHASED

Table 6. **Summary of DoE Energy Savings Calculator Results**

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Application	Industry Group (NAICS Codes)	Type of Savings	2010 Savings (TBtu)	2015 Savings (TBtu)	2020 Savings (TBtu)
Ethylene Production	324110	Upset Avoidance & Process Improvement	2.14	10.26	18.25
Metals Processing	331, 332	Waste Avoidance	2.45	6.96	16.20
Hydrogen Production	325	Process Improvement	0.52	2.73	5.35
TiO_2 Drying	325	Process Improvement	0.01	0.04	0.06
Fired Heaters	324110	Process Improvement	2.14	10.26	18.15
Aluminum Production	3313	Process Improvement	0.39	1.12	2.61
Pulp Production	322110	Process Improvement	0.45	2.06	3.49
TOTAL			8.10	33.42	64.10
Petroleum Refining	324110	Upset Avoidance & Process Improvement	18.59	86.48	149.36
Metals	331, 332	Waste Avoidance	4.41	12.54	29.20
Chemicals	325	Process Improvement	7.95	38.13	67.83
Pulp Mills	322110	Process Improvement	1.06	4.84	8.19
TOTAL			32.01	141.99	254.58
% of 1998 TOTAL IND. CONSUMPTION			0.13%	0.60%	1.07%

Programmatic Merit of PHASED

- **Economic and environmental benefits**
  - Cost reduction: NeSSI+Microanalytics: **>35% life cycle cost savings***

    * J. J. Gunnell and P. vanVuuren, IFPAC-2000 paper **
  - Emissions reductions: for C, NOx, CO, VOC, PM were obtained from the **DOE Energy Calculator****

    ** U.Bonne et al, "NeSSI-Compatible Microanalytics for Process Control Solutions," Honeywell and Consortium, proposal to DOE-S&C, 16-Apr.-03

Programmatic Merit of PHASED

Table 7. Summary of Emissions Reductions from DoE Energy Savings Calculator

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Application	Industry Group (NAICS Codes)	Type of Savings	2010 Emissions Gains Carbon (MMTCE/yr)	NOX (MMlbs/yr)	CO (MMlbs/yr)	VOCS (MMlbs/yr)	Particulates (MMlbs/yr)
Ethylene Production	324110	Upset Avoidance & Process Improvement	0.036	0.629	0.118	0.012	0.011
Metals Processing	331, 332	Waste Avoidance	0.037	0.576	0.154	0.015	0.000
Hydrogen Production	325	Process Improvement	0.008	0.137	0.030	0.003	0.001
TiO_2 Drying	325	Process Improvement	0.000	0.002	0.001	0.000	0.000
Fired Heaters	324110	Process Improvement	0.036	0.630	0.118	0.012	0.011
Aluminum Production	3313	Process Improvement	0.007	0.139	0.019	0.002	0.004
Pulp Production	322110	Process Improvement	0.008	0.138	0.021	0.003	0.004
TOTAL			0.133	2.251	0.460	0.046	0.030
Petroleum Refining	324110	Upset Avoidance & Process Improvement	0.351	5.898	0.744	0.131	0.209
Metals	331, 332	Waste Avoidance	0.066	1.037	0.278	0.027	0.000
Chemicals	325	Process Improvement	0.127	2.110	0.469	0.046	0.020
Pulp Mills	322110	Process Improvement	0.019	0.325	0.050	0.007	0.009
TOTAL			0.563	9.370	1.540	0.210	0.238

Application	Industry Group (NAICS Codes)	Type of Savings	2020 Emissions Gains Carbon (MMTCE/yr)	NOX (MMlbs/yr)	CO (MMlbs/yr)	VOCS (MMlbs/yr)	Particulates (MMlbs/yr)
Ethylene Production	324110	Upset Avoidance & Process Improvement	0.310	5.373	1.003	0.100	0.091
Metals Processing	331, 332	Waste Avoidance	0.243	0.381	1.020	0.097	0.000
Hydrogen Production	325	Process Improvement	0.086	1.419	0.315	0.031	0.014
TiO_2 Drying	325	Process Improvement	0.001	0.015	0.004	0.000	0.000
Fired Heaters	324110	Process Improvement	0.310	5.373	1.003	0.100	0.091
Aluminum Production	3313	Process Improvement	0.050	0.922	0.122	0.013	0.026
Pulp Production	322110	Process Improvement	0.063	1.069	0.164	0.022	0.029
TOTAL			1.063	14.552	3.632	0.364	0.251
Petroleum Refining	324110	Upset Avoidance & Process Improvement	2.898	47.262	6.061	1.046	1.644
Metals	331, 332	Waste Avoidance	0.436	6.863	1.840	0.175	0.000
Chemicals	325	Process Improvement	1.087	17.993	3.998	0.390	0.173
Pulp Mills	322110	Process Improvement	0.147	2.513	0.385	0.052	0.068
TOTAL			4.568	74.631	12.283	1.663	1.885

Summary of PHASED Project

- **Completed the design and fabrication or 1st device**
  - Chip
  - Electronics
  - Software
- **Demonstrated its fundamental feasibility**
- **Initiated its performance characterization**
  - 1-2 seconds per analysis
  - 8 ms peak half-width for 5 ms pulses per heater of multi-stage PC
  - Synchronization between heater wave and sample flow
- **Established field test sites and commercializ. plan**
- **Estimated PHASED/NeSSI energy & emissions saving**
- **Submitted Final Report w/recommended future work**
- **Submitted Proposal to DOE to achieve Future Goals, which also quantifies energy & emissions reductions**